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SPECIFICATION FOR CALCIUM CARBONATE, PRECIPITATED FOR COSMETIC INDUSTRY
(Second Revision)

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Gr 6 *July* 1986

Indian Standard

SPECIFICATION FOR
CALCIUM CARBONATE, PRECIPITATED FOR COSMETIC INDUSTRY

(Second Revision)

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AMENDMENT NO. 1 SEPTEMBER 1998
TO
IS 918 : 1985 SPECIFICATION FOR CALCIUM CARBONATE,PRECIPITATED FOR COSMETIC INDUSTRY

(*Second Revision*)

(*Page* 16, *clause* A-10.0) — Substitute the following for the existing:

"**A-10.0 General** — Two methods have been specified for the determination o heavy metals. In 'Method A' the colour produced with hydrogen sulphid solution is compared with that obtained with standard lead solution while i 'Method B', the colour produced with thioacetamide reagent is compared witl that obtained with standard lead solution."

(*Page* 16, *clause* A-10.0) — Insert 'METHOD A' after clause A-10.0.

(*Page* 16, *clause* A-10.3.1) — Insert 'METHOD B' after clause A-10.3.1:

METHOD B

A-10.4 Apparatus

A-10.4.1 *Weighing Balance* — 0.000 1 g accuracy.

A-10.4.2 *Nessler Cylinders* — 25/50 ml.

A-10.4.3 *Measuring Cylinder* — 10 ml, 25 ml.

A-10.4.4 *Pipette* — 10 ml, 2 ml.

A-10.4.5 *Volumetric Flask* — 100 ml.

A-10.4.6 *Evaporating Dish*

A-10.5 Reagents

A-10.5.1 *Acetate Buffer,pH* — 3.5.

Dissolve 25 g of ammonium acetate in 25 ml of water and add 38 ml of 7M hydrochloric acid. Adjust the *p*H to 3.5 with either 2M hydrochloric acid or 6M ammonia and dilute to 100 ml with water.

A-10.5.2 *Glycerol Mixture* — To 15 ml of sodium hydroxide, 1M, add 5 ml o water and 20 ml of 85 percent glycerol. Mix well.

A-10.5.3 *Thioacetamide Reagent* — Weigh 80 mg of thioacetamide and add to it 2 ml of water. Shake to dissolve. Add to it 10 ml glycerol mixture, heat in water bath for 20 seconds, cool and use immediately.

A-10.5.4 *Acetic Acid Solution* — 6M

342 ml of glacial acetic acid with sufficient water to produce 1 000 ml.

A-10.5.5 *Lead Nitrate (Stock) Solution* — Dissolve 0.159 8 g of lead nitrate in 100 ml of water to which 1 ml of nitric acid has been added. Dilute to 1 000 ml.

A-10.5.6 *Standard Lead Solution* — (10 ppm/ml).

Dilute 10 ml of lead nitrate stock solution with water to 100 ml. Each ml is equivalent to 10 ppm of lead.

A-10.6 Procedure

Into 50-ml Nessler cylinder, pipette 1 ml of standard lead solution and dilute with water to 25 ml, add 2 ml acetate buffer (*p*H 3.5), mix. Add 1.2 ml of thioacetamide reagent, mix immediately. Dilute with water to 50 ml and allow to stand for 2 minutes.

Dissolve 1 g of sample in 25 ml of 6M acetic acid. When effervescence ceases, boil the solution for 2 minutes, cool, filter into Nessler cylinder. Add 2 ml of acetate buffer (*p*H 3.5), mix. Add 1.2 ml of thioacetamide reagent, mix immediately. Dilute with water to 50 ml, allow to stand for 2 minutes.

A-10.6.1 Any brown colour produced is not more intense than that produced in the standard solution.

(PCD 19)

Printed at Simco Printing Press, Delhi

Indian Standard

SPECIFICATION FOR CALCIUM CARBONATE, PRECIPITATED FOR COSMETIC INDUSTRY

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 11 December 1985, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1958 and subsequently revised in 1968. The Sectional Committee responsible for this standard reviewed it in the light of development of the cosmetics industry in this country and decided to revise it in order to bring the standard up to the requirements of the industry. In this revision, the requirement soluble alkali content (as Na_2O) has been changed to *p*H of 10 percent aqueous suspension in order to determine the alkalinity more accurately. Due to strong reservations regarding the use of potassium cyanide in a cosmetic testing laboratory in this revision, potassium cyanide method for test of heavy metals has been replaced by hydrogen sulphide method. All other changes considered necessary to align the standard with other Indian Standards on raw materials for cosmetics have also been included in this standard. Information on the two crystalline phases of calcium carbonate, namely, calcite and aragonite, has been introduced for the information of the users of this standard in Appendix C.

0.3 Precipitated calcium carbonate is used in the cosmetic industry in the manufacture of tooth pastes, solid dentifrices, tooth powders, face powders and talcum powders.

0.4 In the cosmetic industry, it is necessary that precipitated calcium carbonate is free from abrasive and gritty impurities as far as possible, since their presence will cause damage to the epidermis, to the enamel of the teeth and also cause scratching of the gums.

0.5 For particle size, the use of IS-sieves [conforming to IS : 460 (Part 1)-1978*] has been prescribed. Where IS-sieves are not available, other equivalent sieves, as judged by the aperture size, may be used.

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*Specification for test sieves : Part 1 Wire cloth test sieves (*second revision*).

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling. and test for precipitated calcium carbonate for cosmetic industry.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of fine, white microcrystalline powder without any taste or odour. It shall be stable in air.

2.1.1 The material exists in two crystalline phases, namely, calcite and aragonite.

2.2 The material, when tested according to the methods prescribed in Appendix A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

3. PACKING AND MARKING

3.1 The material shall be packed as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and legibly marked with the following information:

a) Name and mass of the material in the container;

b) Manufacturer's name and recognised trade-mark, if any;

c) Batch number in code or otherwise to enable the lot of manufacture to be traced back from records; and

d) Date of manufacture.

---

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR PRECIPITATED CALCIUM CARBONATE FOR COSMETIC INDUSTRY

(*Clause* 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Fineness:		
	a) Residue on 75-µm IS-sieve, percent by mass, *Max*	0·5	A-2
	b) Residue on 150-µm IS-sieve, percent by mass, *Max*	0·1	
ii)	Loss on drying, percent by mass, *Max*	1·0	A-3
iii)	Bulk density, g/ml	As agrede to between the purchaser and the supplier	A-4
iv)	Calcium carbonate (as $CaCO_3$), percent by mass (on dry basis), *Min*	98·0	A-5
v)	*Magnesium and alkali salts, percent by mass, *Max*	1·0	A-6
vi)	Aluminium, iron, phosphates and matter insoluble in hydrochloric acid, percent by mass, *Max*	0·5	A-7
vii)	Iron (as Fe), percent by mass, *Max*	0·03	A-8
viii)	Arsenic (as As_2O_3), parts per million, *Max*	2	A-9
ix)	Heavy metals (as Pb), parts per million, *Max*	10	A-10
x)	Chlorides (as Cl), percent by mass, *Max*	0·04	A-11
xi)	*p*H of 10 percent aqueous suspension	9·0 to 10·0	A-12
xii)	Barium	pass test	A-13
xiii)	Sulphates	pass test	A-14
xiv)	*Flow point	agreed to tween the ırchaser and supplier	A-15

*Applicable only for use in the manufacture of tooth paste.

3.3 The product may also be marked with Standard mark.

3.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method for preparing representative test samples of the material and the criterion for conformity shall be as given in Appendix B

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR CALCIUM CARBONATE, PRECIPITATED, FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals (*see* Note) and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contail impurities which affect the results of analysis.

A-2. DETERMINATION OF FINENESS

A-2.1 Apparatus

A-2.1.1 *Gallie-Porritt Sieve* — The apparatus (*see* Fig. 1) consists of a metal funnel terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, a wire gauze (conforming to 150-μm IS-sieve or 75-μm IS-sieve, as appropriate) of 25 mm diameter is soldered. Water under pressure of 1·5 to 3·5 kg/cm^2 is supplied by a tube fitted with a nozzle designed to discharge a spreading jet throughout the wire gauze, and the tube is so arranged that the distance of the orifice in relation to the wire gauze can be adjusted. The tube is provided with a filter to

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*Specification for water for general laboratory use (*second revision*).

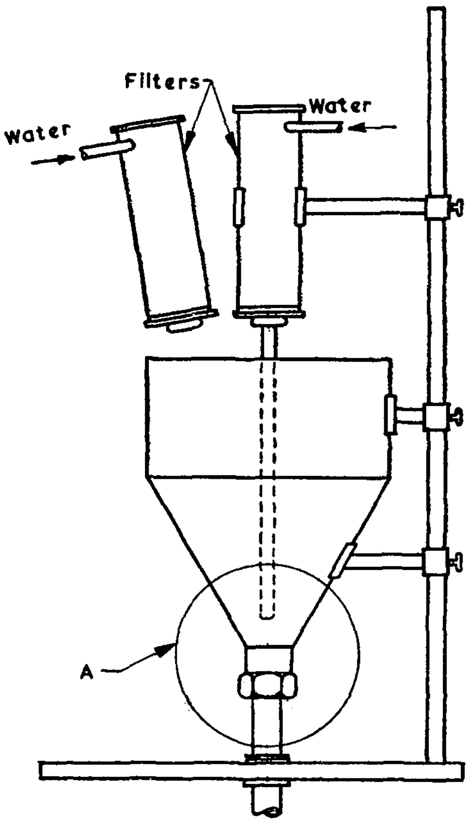
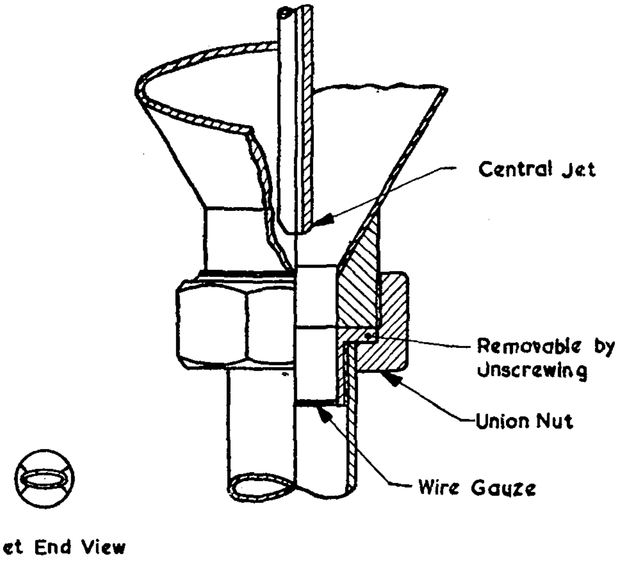


FIG. 1



ENLARGED VIEW AT A

GALLIE-PORRITT SIEVE

ensure removal of any solid particles from the water. A second tube which is also provided with a filter is improvised to supply a gentle stream of a water for wetting the powder and for supplying more water to the funnel during the progress of the test.

A-2.1.1.1 The area of the wire gauze shall be sufficiently large to prevent undue loss of pressure head in the case of the first tube.

A-2.2 Procedure

A-2.2.1 Carry out the test separately using 150-µm IS-sieve and 75-µm IS-sieve.

A-2.2.2 Weigh accurately 50 ± 1 g of the material. Half fill the funnel with water from the subsidiary supply, then bring the central jet (*see* Fig. 1) into operation. Transfer the weighed material to the metal funnel either dry or in the form of a slurry with a suitable wetting agent. Turn off the subsidiary supply and, if necessary, adjust the height of the central jet so that the level of water in the funnel commences to fall. When the level has fallen within 50 mm of the sieve, turn on subsidiary supply until the level is within 50 mm from the top of the funnel and maintain there until the liquid appears clear and then for a further period of one minute. Turn off the subsidiary supply and allow the level to fall again. When the funnel is empty, turn off the main jet and allow to drain. Remove the cup and examine for any large soft agglomerates. If present, break them up gently with the finger and replace the cup and wash with water from the subsidiary supply for 2 minutes. Transfer the residue on the sieve to a watch glass and dry to constant mass at 105°C. Note the lowest mass.

A-2.2.3 Powders which are very light are difficult to be wetted at a time in large quantity of 50 g. To facilitate wetting of these powders, either a small amount of wetting agent may be added or the material should be added in small quantities into the apparatus.

A-2.3 Calculation

A-2.3.1 From the mass of the residue in the sieves, calculate the percentage by mass of the residue on each sieve.

A-3. DETERMINATION OF LOSS ON DRYING

A-3.1 Procedure — Weigh accurately about 5 g of the material in a weighed, clean and dry squat form weighing bottle, and dry to constant mass at 105°C ± 2°C.

A-3.2 Calculation

$$\text{Loss on drying, percent by mass} = 100 \frac{W_1}{W_2}$$

where

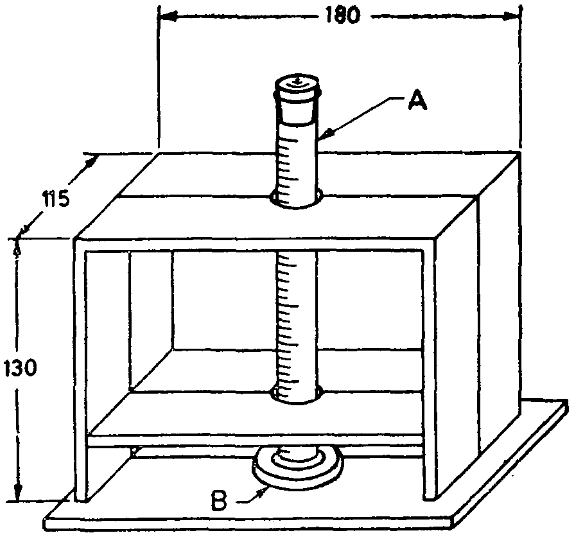
W_1 = loss in mass g on drying, and

W_2 = mass in g of the material taken for the test.

A-4. DETERMINATION OF BULK DENSITY

A-4.1 Apparatus

A-4.1.1 The apparatus is shown in Fig. 2. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error of less than one millilitre. The distance between zero and 250-ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 mm. The distance between the flat ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 25 ± 2 mm.



All dimensions in millimetres.

FIG. 2 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-4.1.2 *Balance* — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0·1 g.

A-4.1.3 *Rubber Base Pad B* — It shall have a shore hardness of 35 to 50.

A-4.2 Procedure — Weigh about 42 g of the material on a piece of paper. Fold a piece of black glazed paper (25 × 25 cm) with two parallel creases to form a channel 12·5 mm down the middle of the paper. Counterpoise the paper on the balance, then place it flat on the bench. Place a 500 µm IS-sieve on the black glazed paper in such a way that there is a 50-mm space between the sieve and the paper. Transfer the weighed material to the sieve and with the back of the finger tips fitted with rubber finger-stalls lightly rub all the powder through the sieve using short strokes. Transfer the black glazed paper with the sieved material to the balance and adjust the weight of the sieved material to 40 g. Pick up the paper and form it into a chute. Allow it to lie between the thumb and fingers on the palm of the hand and introduce it into the cylinder, which should be held at 45° to the vertical. Slip the powder gently and smoothly into the measuring cylinder without knocking or squeezing. Assemble the measuring cylinder as shown in Fig. 2. With the thumb and four fingers of one hand gently grasp the upper part of the cylinder, and within one second lift it as far as 25 mm (*see* Fig. 2). Do not jerk the cylinder by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping until 50 complete drops have been given. Once in every 2 seconds, a gentle twist of about 10° should be given to the cylinder. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-4.3 Calculation

$$\text{Bulk density, g/ml} = \frac{40}{V}$$

where

V = volume in ml of the sieved material.

A-5. DETERMINATION OF CALCIUM CARBONATE

A-5.0 Methods — Two methods have been prescribed for this test, namely, Method A and Method B. Method A is the classical method and Method B is the complexometric method. In case of dispute, Method A shall be followed.

A-5.1 Method A

A-5.1.0 *Outline of the Method* — Calcium is precipitated as its oxalate; the precipitate is dissolved in dilute sulphuric acid and the solution is titrated against permanganate solution.

A-5.1.1 *Reagents*

A-5.1.1.1 *Dilute hydrochloric acid* — Approximately 5 N.

A-5.1.1.2 *Ammonium oxalate solution* — Saturated.

A-5.1.1.3 *Dilute ammonium hydroxide* — Approximately 5 N and 1 N.

A-5.1.1.4 *Dilute potassium permanganate solution* — Approximately 0·01 N.

A-5.1.1.5 *Dilute sulphuric acid* — Approximately 5 N.

A-5.1.1.6 *Standard potassium permanganate solution* — 0·1 N.

A-5.1.2 *Procedure* — Weigh accurately about one gram of the material dried as in **A-3.1**, and transfer into a 250-ml beaker. Moisten thoroughly with a few millilitres of water and add dilute hydrochloric acid dropwise until solution is complete. Continue adding the acid until a total of 40 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer completely to a 200-ml graduated flask, add water to make up the volume and mix thoroughly. Pipette out 50 ml of this solution to a beaker and add 75 ml of water. Heat almost to boiling and add 15 ml of ammonium oxalate solution. To the hot solution, constantly stirred, add dilute ammonium hydroxide (5 N) dropwise until the mixture is strongly alkaline. Let it stand for two hours. Decant the clear supernatant liquid through a filter paper with cold water. Wash the precipitate first with dilute ammonium hydroxide (1 N) and then with cold water until the filtrate does not decolorize hot dilute potassium permanganate solution in presence of dilute sulphuric acid. Puncture the filter paper and transfer the precipitate to a beaker with a fine jet of hot water. Add about 30 ml of dilute sulphuric acid, heat at 60°C and titrate with standard potassium permanganate solution. Towards end of the titration introduce the filter paper, which was used for filtration, into the titration vessel and carry out the titration till the end point is reached. Carry out a blank test with all reagents but without the material following the procedure for the test.

A-5.1.3 *Calculation*

$$\text{Calcium carbonate (as } CaCO_3 \text{), percent by mass} = \frac{20{\cdot}02\,(V_1 - V_2)}{W}$$

where

V_1 = volume in ml of standard potassium permanganate solution used for the test,

V_2 = volume in ml of standard potassium permanganate solution used for the blank test,

N = normality of standard potassium permanganate solution, and

W = mass in g of the material taken for the test.

A-5.2 Method B

A-5.2.0 *Outline of the Method* — The solution of the material, after adjustment of *p*H, is titrated with EDTA solution using murexide indicator.

A-5.2.1 *Reagents*

A-5.2.1.1 *Dilute hydrochloric acid* — Approximately 5 N.

A-5.2.1.2 *Dilute sodium hydroxide solution* — Approximately 1 N.

A-5.2.1.3 *Standard calcium carbonate solution* — 0·01 N. Weigh 0·500 4 g of calcium carbonate dried as in **A-3.1**, and dissolve in the minimum quantity of dilute hydrochloric acid. Make up the volume with water to 1 litre.

A-5.2.1.4 *Murexide indicator* — Triturate 0·2 g of murexide with 50 g of potassium chloride until a homogeneous mixture is obtained.

A-5.2.1.5 *Standard disodium ethylenediamine tetraacetate (EDTA) solution* — Weigh 3·7 g of disodium ethylenediamine tetraacetate dehydrate and dissolve in water. Make up the volume to 1 litre. Standardize the solution against standard calcium carbonate solution as in **A-5.2.3**.

A-5.2.2 *Prepared Sample Solution* — Weigh accurately about 3 g of the material, dried as in **A-3.1**, and transfer it to a beaker. Moisten thoroughly with a few millilitres of water and add dilute hydrochloric acid dropwise until the solution is complete. Continue adding the acid until a total of 50 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer quantitatively to a 500-ml graduated flask. Pipette out 100 ml of this solution and dilute further to 1 litre. Use this diluted solution for the tests in **A-5.2.3** and **A-6.2**.

A-5.2.3 *Procedure* — Pipette out 10 ml of the *prepared sample solution* (**A-5.2.2**) into a 250-ml conical flask. Add 25 ml of water and 10 ml of dilute sodium hydroxide solution (*p*H of mixture should be greater than 12). Add 0·3 g of murexide indicator and titrate with standard EDTA solution until the colour changes from red to distinct blue violet.

A-5.2.4 *Calculation*

$$\text{Calcium carbonote (as CaCO}_3\text{), percent by mass} = \frac{2{\cdot}502\,VN}{W}$$

where

V = volume in ml of standard EDTA solution,

N = normality of standard EDTA solution, and

W = mass in g of the material taken for the test in **A-5.2.2**.

A-6. TEST FOR MAGNESIUM AND ALKALI SALTS

A-6.0 Outline of the Method — The solution obtained after precipitation of oxalates in ammonical medium is evaporated to dryness, and the residue obtained is ignited and weighed.

A-6.1 Reagents

A-6.1.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-6.1.2 *Oxalic Acid Solution* — 6 percent.

A-6.1.3 *Methyl Red Indicator Solution* — Dissolve 0·03 g of methyl red in 100 ml of water.

A-6.1.4 *Ammonium Hydroxide* — (1 : 1) (*v/v*).

A-6.1.5 *Concentrated Sulphuric Acid* — *See* IS : 266-1977†.

A-6.2 Procedure — Weigh accurately about 1 g of the material in a beaker, add to it 40 ml of water and 5 ml of concentrated hydrochloric acid. Heat and then boil for 1 minute. To the hot solution add rapidly 40 ml of oxalic acid solution and stir vigorously until precipitation is well established. Add immediately to the warm mixture 2 drops of methyl red indicator and then add dropwise ammonium hydroxide until the mixture is just alkaline. Cool to room temperature, transfer to 100-ml graduated cylinder, dilute with water to 100 ml, mix and let stand for 4 hours. Filter, take 50 ml of the filtrate in a weighed platinum dish and add to it 0·5 ml of concentrated sulphuric acid. Evaporate the mixture on a steam bath to a small volume. Then carefully heat over a small flame to dryness and then continue heating to complete decomposition and volatilization of ammonium salts. Cool in a desiccator and weigh. Repeat heating, cooling and weighing until constant mass (±1 mg) is obtained.

A-6.3 The material shall be taken to have passed the test if the mass of the residue does not exceed 5 mg.

A-7. DETERMINATION OF ALUMINIUM IRON PHOSPHATES AND MATTER INSOLUBLE IN HYDROCHLORIC ACID

A-7.0 Outline of the Method — The material is boiled with hydrochloric acid and the solution along with any insoluble matter is made alkaline with ammonia. The total residue is then weighed.

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — Reagent grade.

A-7.1.2 *Dilute Ammonium Hydroxide* — Approximately 5 N.

A-7.1.3 *Methyl Red Indicator* — Same as in **A-6.1.3**.

A-7.1.4 *Ammonium Chloride Solution* — Approximately 2 percent.

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*Specification for hydrochloric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

A-7.1.5 *Dilute Hydrochloric Acid* — Approximately 5 N.

A-7.1.6 *Ammonium Nitrate Solution* — Approximately 2 percent.

A-7.2 Procedure — Weigh accurately about 2 g of the material, dissolve in a mixture of 15 to 20 drops of concentrated nitric acid and 10 ml of concentrated hydrochloric acid and 75 ml of water; boil; cool and make alkaline with dilute ammonium hydroxide using methyl red indicator. Boil again for one minute, filter and wash the precipitate with hot ammonium chloride solution. Dissolve the precipitate as completely as possible by passing 20 ml of hot dilute hydrochloric acid through the filter paper and wash the filter paper with sufficient hot water to adjust the volume of the solution to 50 ml. Boil the solution, cool and make alkaline with dilute ammonium hydroxide using methyl red indicator. Boil again for one minute, filter through the same filter paper and wash the precipitate with hot ammonium nitrate solution. Place the filter paper with its contents in a platinum crucible. Dry in an air-oven, char the filter paper by gentle heating over a low flame and then slowly increase the flame and finally ignite to constant mass at 900° to 950°C.

A-7.3 Calculation

$$\text{Aluminium, iron, phosphates and other matter insoluble in hydrochloride acid, percent by mass} = \frac{100\,A}{W}$$

where

A = mass in g of the ignited residue, and
W = mass in g of the material taken for the test.

A-8. TEST FOR IRON

A-8.0 Outline of the Method — The colour produced with thioglycollic acid in ammonical medium is compared with that obtained with standard iron solution.

A-8.1 Apparatus

A-8.1.1 *Nessler Tubes* — 50-ml capacity.

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid* — Reagent grade.

A-8.2.2 *Citric Acid Solution* —Approximately 20 percent.

A-8.2.3 *Thioglycollic Acid*

A-8.2.4 *Dilute Ammonium Hydroxide* — Approximately 5 N.

A-8.2.5 *Standard Iron Solution* — Dissolve 0·702 g of ammonium ferrous sulphate [$Fe SO_4. (NH_4)_2SO_4.6H_2O$] in water containing 10 ml of dilute sulphuric acid. Transfer to a 1 000 ml graduated flask and dilute with water to the mark. Pipette out 10 ml of this solution into a 100 ml graduated flask and dilute this with water to the mark. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-8.3 Procedure — Dissolve 1·000 g of the material in a mixture of 25 ml of water and 2·5 ml of concentrated hydrochloric acid. Boil, dilute with water and make up the volume in a volumetric flask to 200 ml. Transfer by pipette 40 ml of this solution to a Nessler tube. Add 2 ml of citric acid solution and 2 drops of thioglycollic acid. Mix and make the solution alkaline with dilute ammonium hydroxide. Make up the volume to 50 ml mark. Carry out a control test in another Nessler tube using 6 ml of standard iron solution in place of the material and the same quantities of other reagents. Compare the colour produced, if any, after 5 minutes.

A-8.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-9. TEST FOR ARSENIC

A-9.0 Outline of the Method — The stain obtained on mercuric bromide paper by arsine from the material is compared with a standard stain.

A-9.1 Reagents

A-9.1.1 *Concentrated Hydrochloric Acid* — Reagent grade.

A-9.1.2 *Brominated Hydrochloric Acid* — Shake 1 ml of bromine with 100 ml of concentrated hydrochloric acid.

A-9.1.3 *Stannous Chloride Solution* — Dissolve 80 g of stannous chloride in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

A-9.1.4 *Mixed Acid* — Dilute one volume of concentrated sulphuric acid (reagent grade) with four volumes of water, and to this add 10 g of sodium chloride for each 100 ml of solution.

A-9.1.5 *Ferric Ammonium Sulphate Solution* — Dissolve 84 g of ferric ammonium sulphate in water with 10 ml of mixed acid and make up to one litre.

A-9.2 Procedure — Dissolve 1·000 g of the material in 6 ml of brominated hydrochloric acid and 20 ml of water. Remove the excess of bromine with a few drops of stannous chloride solution. Add water to make up the volume to about 50 ml. Transfer the solution to the Gutzeit bottle, add 2 ml of ferric ammonium sulphate solution and carry out the test as prescribed in IS : 2088-1983*, using 0·002 mg of arsenic trioxide for the comparison stain.

A-10. TEST FOR HEAVY METALS

A-10.0 Outline of the Method — The colour produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50-ml capacity.

A-10.2 Reagents

A-10.2.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976†.

A-10.2.2 *Concentrated Nitric Acid* — *See* IS : 264-1976‡.

A-10.2.3 *Dilute Acetic Acid* — 6 percent acetic acid (60 ml diluted to 1 000 ml with water).

A-10.2.4 *Hydrogen Sulphide Solution* — Saturated.

A-10.2.5 *Standard Lead Solution* — Dissolve 1·600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-10.3 Procedure — Ignite 2·000 g of the material in silica dish until completely ashed. To the residue add 1 ml of hydrochloric acid and 0·5 ml nitric acid and evaporate to dryness on the steam bath. Dissolve the residue in 5 ml hot water and 1 ml dilute acetic acid and transfer to Nessler cylinder washing in with water. In the second Nessler cylinder, place 1 ml of dilute acetic acid and 2 ml of standard lead solution. Add to each cylinder 10 ml of hydrogen sulphide solution and make up the volume with water to 50 ml. Mix, allow to stand for 10 minutes and then compare the colour produced in the two Nessler cylinders.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the second Nessler cylinder.

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*Method for determination of arsenic (*second revision*).

†Specification for hydrochloric acid (*second revision*).

‡Specification for nitric acid (*second revision*).

A-11. TEST FOR CHLORIDES

A-11.0 Outline of the Method — The turbidity produced with silver nitrate solution is matched against that obtained with standard chloride solution.

A-11.1 Apparatus

A-11.1.1 *Nessler Tubes* — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 *Concentrated Nitric Acid* — Reagent grade.

A-11.2.2 *Standard Sodium Chloride Solution* — Dissolve 1·649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of the solution, dilute with water and make up the solution to 1 000 ml. One millilitre of this solution contains 0·01 mg of chloride (as Cl).

A-11.2.3 *Silver Nitrate Solution* — Appoximately 2 percent (*m/v*).

A-11.3 Procedure — Dissolve 1·000 g of the material in water by the addition of 3 ml of concentrated nitric acid, boil cautiously to remove carbon dioxide, cool and make up the volume to 100 ml. Pipette out 10 ml of this solution into a Nessler tube, and add one millilitre of silver nitrate solution. Carry out a control test in the other Nessler tube using 4 ml of standard sodium chloride solution and the same quantities of other reagents. Stir both the solutions with glass rods and compare the turbidity produced in the two tubes after 5 minutes.

A-11.3.1 The limit prescribed shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-12. DETERMINATION OF *p*H OF AQUEOUS SUSPENSION

A-12.1 Procedure — Accurately weigh 10·0 g of the test substance in a beaker, and add about 80 ml of carbon dioxide free water, stir to make a slurry and transfer quantitatively into a 100 ml volumetric flask. Add more of carbon dioxide free water and make up to volume. Shake and transfer into a 250-ml standard ground glass joint conical flask, attach a water condenser and reflux for 10 minutes. Cool and determine the *p*H of this suspension using a suitable *p*H meter.

A-13. TEST FOR BARIUM

A-13.1 Reagent

A-13.1.1 *Concentrated Hydrochloric Acid* — Same as in **A-8.2.1.**

A-13.2 Procedure — Dip a clean platinum wire into the material. Moisten it with a drop of concentrated hydrochloric acid and hold it in a non-luminous flame.

A-13.2.1 The material shall be considered to have passed the test if no yellowish-green colour is imparted to the flame.

A-14. TEST FOR SULPHATES

A-14.0 Outline of the Method — The turbidity produced with barium chloride solution is compared with that obtained with a standard sulphate solution.

A-14.1 Apparatus

A-14.1.1 *Nessler Tubes* — 50-ml capacity.

A-14.2 Reagents

A-14.2.1 *Dilute Hydrochloric Acid* — Approximately 5 N.

A-14.2.2 *Standard Sulphuric Acid* — 0·01 N.

A-14.2.3 *Barium Chloride Solution* — Approximately 10 percent by mass.

A-14.3 Procedure — Dissolve 0·50 g of the material in water by addition of 3 ml of dilute hydrochloric acid and boil to remove carbon dioxide. Transfer the solution to a Nessler tube and add water to make up the volume to 50 ml. Add 1 ml of barium chloride solution and stir the mixture. Carry out a control test in the other Nessler tube using 2·5 ml of standard sulphuric acid and the same quantities of other reagents.

A-14.3.1 The material shall be considered to have passed the test if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-15. DETERMINATION OF FLOW POINT

A-15.0 Outline of the Method — Measured increments of water are added to the material in a test tube and the quantity of water required to make the paste flow is determined.

A-15.1 Procedure — From a 50-ml burette, introduce a volume of water equal to about four-fifths of the expected flow point into a thick-walled test tube (internal dimensions 20 × 150 mm) fitted with a rubber bung. Weigh 15·0 g of the material on to a sheet of black glazed paper and add a little at a time to the test tube. After each addition, replace the bung and shake vigorously. When the paste gets stiff and does not rattle on shaking, add further quantities of water in 1-ml portions alternately with a portion of the remaining material until all of the material is in the test tube and has formed a paste of stiff consistency. Now add water in 0·5 ml portions with vigorous shaking between additions (shaking may be test done by pounding over rubber pad or folded duster). To test when the flow point has been reached, give the test tube a few gentle taps on the bench to shake the paste to the bottom and then invert at an angle of about 15° from the vertical and slowly rotate (do not give the tube any movement to force or shake out the paste). After five seconds, find out whether the paste flows out evenly to the other end of the tube. Note the total volume of water used when this stage is reached.

NOTE — The flow should not be sluggish and the slurry should be free from lumbs. If the test tube is rotated slowly at the flow point in a horizontal position the slurry should flow down the walls and should not rotate with the test tube.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF CALCIUM CARBONATE, PRECIPITATED FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw representative samples, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a stopper after filling, and marked with full details of sampling, the date of sampling, and the year of manufacture of the material.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal atmospheric temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested for each lot separately for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number of container (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size (N) (1)	No. of Containers to be Chosen (n) (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 Those containers to be selected for sampling shall be chosen at random from the lot till requisite sample is obtained and for this purpose random number tables shall be used. In case such tables are not available, the following procedure shall be adopted:

> Starting from any container, count them 1, 2, 3......, etc, up to r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see*

Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in **2** and shall be not less than 200 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken for each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 500 g. This composite sample shall be divided into three equal parts; one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

B-4. NUMBER OF TEST

B-4.1 Tests for all the requirements given in **2** shall be conducted on the composite sample.

B-5. CRITERION FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to the specification if the composite sample satisfies all the requirements given in **2**.

APPENDIX C

(*Clause* 0.2)

INFORMATION ON CRYSTALLINE PHASES OF CALCIUM CARBONATE

C-1. PRECIPITATED CALCIUM CARBONATE

C-1.1 This material is obtained in two crystalline forms, namely, calcite and aragonite. Important properties of the two forms along with their comparative importance for tooth paste formulation are given below.

Sl. No.	*Characteristic*	*Crystalline Form*	
		Calcite	Aragonite
(1)	(2)	(3)	(4)
i)	Crystalline form	Rhombohedral Hexagonal Crystals or Plates	Ortho-rhombic Needle-Shaped or Hairlike Crystals
ii)	Flow point, ml	21 to 24	20
iii)	Hardness (basis-dental enamel=5)	3·0	3·5 to 4·0
iv)	Solubility in water at 25°C, parts per million	140	150

C-1.2 Aragonitic chalks are preferred in tooth paste formulation because of their higher abrading (that is, cleaning) power than calcitic chalks of similar particle size. However, if less abrasive pastes are required, it is preferable to use calcitic rather than aragonitic chalks.

C-1.3 For the same particle size, calcitic chalks have a larger surface area, that is, higher absorbency than aragonitic chalks and, as such, a lower concentration of calcitic chalk will be required in the tooth paste formulation than that of aragonitic chalk.

C-1.4 The proportion of aragonite and calcite forms of chalk can be estimated by the Meigen test, which employs the greater solubility of the aragonite form to precipitate blue cobalt hydroxide from a solution of cobalt nitrate on to the surface of the suspended powder. The colour of the suspension or the filtered solid is compared with a series of standards. The method originally described by M. C. Smith in the Journal of the Society of Chemical Industry (1937, 16, 283T-287T), is given in **C-2**.

C-2. METHOD FOR ESTIMATING THE TWO PHASES

C-2.1 Procedure — Transfer about 0·2 g of the material, accurately weighed, to a 150-ml beaker, add 50 ml of water and a drop of phenolphthalein indicator solution (0·1 percent alcoholic solution) and neutralize the mixture with 0·1 N nitric acid. Heat and when just vigorously boiling, add rapidly 2 ml of 1 N cobalt nitrate solution from a pipette. The suspension froths up about 10 seconds after adding the cobalt reagent, indicating that the heating is sufficiently vigorous. Continue boiling for 30 seconds and cool rapidly by adding 50 ml of ice-cold distilled water. Pour off the pink supernatant liquid and wash the suspensoids once. The decantation is easy because the reaction markedly flocculates the suspension.

C-2.1.1 The colour comparison may be made most accurately with the suspension in a test tube after reducing the volume to 10 ml. Made-up mixtures of calcite and aragonite are used to prepare standards for comparison.

C-2.2 Reproducibility of Results — With the test carried out as in **C-2.1**, the colour is white with calcite and deep purple with aragonite. Reproducibility is 10 percent below 50 percent aragonite, 20 percent with larger proportions and 80 percent aragonite is just distinguishable from the pure form. It is important to ensure uniform suspension during visual comparison.

C-2.3 Interference — Large impurities of soluble chlorides, alkali, phosphates and magnesium affect the results.

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